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Indian Standard

ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF ZINC

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ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF ZINC

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Indian Standard

ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF ZINC

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 September 1981, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is dependent on the fact that metallic elements, in the ground state, will absorb light of the same wavelength they emit when excited. When radiation from a given excited element is passed through a flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms with the flame. A hollow cathode lamp whose cathode is made of the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant-fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation.

0.3 This method covers the determination of zinc in low as well as high concentration, for example, in zinc ash, zinc sulphate, metals, etc. Various products where zinc estimation may be required include fertilizers, soils, food products, zinc smelter, animal feeds, paper pulp, petroleum products, water, electroplating solution, aluminium alloys, magnesium alloys, non-ferrous alloys, high purity metals, plastics, etc.

0.3.1 For a specific method of determination of zinc in zinc sulphate, agricultural grade, reference should be made to IS : 8249-1976* and its Amendment No. 1, November 1980.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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*Specification for zinc sulphate, agricultural grade.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the atomic absorption spectrometric method for the determination of zinc.

2. OUTLINE OF THE METHOD

2.1 Dissolve the sample in hydrochloric acid, oxidize with concentrated nitric acid and then evaporate to dryness. Dissolve again in hydrochloric acid (1 : 1) by boiling, allow to settle and filter. Suitable dilutions are made to aspirate to air-acetylene flame, making standards in the same way. The most sensitive zinc absorption line is at 213·9 nm; however, 307·6 nm line is useful at higher concentrations (1 000 mg/l to 20 000 Mg/l) of zinc. Although most workers use air-acetylene flame, air-propane flame provides a 20 percent improvement. An oxidizing flame is recommended at 213·9 nm. The sensitivity is 0·025 μg/ml in aqueous solution and the detection limit is 0·007 μg/ml. The upper concentration limit is 5μg/ml and by the rotation of the burner the upper limit may be extended to 50μg/ml. Molecular absorption and scattering are appreciable at 213·9 nm and require proper correction.

3. INTERFERENCE

3.1 No chemical interferences have been found in air-acetylene flame At 213·9 nm, non-atomic species in the flame absorb strongly. When the solution has a high concentration of dissolved solids, it is necessary to correct for non-atomic absorption using hydrogen continuum lamp.

3.2 At each level of 1 000 μg/ml, only silicon exhibits a significant effect and lowers the absorption. High concentration of gold interferes slightly. Perchloric acid and sulphuric acid cause a slight depression in the absorption. Equivalent amount of acids should be added to standards.

3.3 Copper and phosphorus (100μg/ml) interfere, the former enhancing and the latter depressing the zinc absorption. Addition of 1 000μg/ml strontium checks the interference due to these elements.

3.4 Zinc adsorption is constant in the *p*H range of 2 to 5. Between 5 to 10*p*H range, [redacted] a sharp decrease in adsorption is observed. At *p*H above 10, zinc adsorption levels off to a constant value. Therefore, the *p*H of the solution and the standards should be the same.

4. APPARATUS

4.1 Atomic Absorption Spectrophotometer — generally with the following parameters.

4.1.1 *Lamp Current*—5 m A.

4.1.2 *Support* — air.

4.1.3 *Fuel* — acetylene.

4.1.4 *Flame Stoichiometry* — Oxidizing.

4.1.5 *Wavelength and Working Range*

Wavelength, nm	*Band Pass, nm*	*Working Range* μg/ml
213·9	0·2	0·4 — 1·6
307·6	0·5	3 500 — 14 000

By rotation of burner (by reducing the path length of the sample) a 213·9 nm measurement can be taken up to 50 μg/ml.

5. REAGENTS

5.1 Zinc Metal — pure.

5.2 Concentrated Hydrochloric Acid — *See* IS : 265-1976*.

5.3 Concentrated Nitric Acid — *See* IS : 264 - 1976†.

6. PROCEDURE

6.1 Transfer an appropriate quantity of the sample to a 400-ml beaker and add 50 ml of concentrated hydrochloric acid slowly. Heat to dissolve, and oxidize with drop-by-drop addition of concentrated nitric acid. Evaporate to dryness. Dissolve again in 40 ml of hydrochloric acid (1 : 1) and heat to boiling. Add 100 ml of distilled water, boil again, cool and filter to remove siliceous matter and make up to 250 ml with distilled water. Make appropriate dilutions.

NOTE — In the case of food organic substances a representative sample estimated to contain 25-100 μg of zinc is accurately weighed into a clean platinum dish and ashed at temperature up to 525°C and is dissolved in minimum volume of HCl (1+1). Add 20 ml water and evaporate to dryness on steam bath. Add 20 ml 0·1 *N* HCl. Heat for 5 min and filter with several portions of 0·1 *N* HCl. Further dilution may be made if required to attain the working range of the instrument.

6.2 Optimize the response of the instrument by adjusting burner height and flame. Aspirate distilled water to get zero absorption. Aspirate blank and note down absorption, if any. Again aspirate distilled water to check zero. When stable response is observed, aspirate standards (at least 4) and note down the absorption. Aspirate the sample to get its absorption. Prepare calibration curve by plotting the net absorption values of the standards against concentration in mg/l of zinc. Locate the point of sample absorbance and calculate the concentration of zinc in the sample.

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*Specification for hydrochloric acid (*second revision*).

†Specification for nitric acid (*second revision*).

7. CALCULATION

7.1 Zinc, percent by mass $= \frac{C \times V}{10^6} \times \frac{100}{M}$

where

C = Concentration of zinc in µg/ml in the final solution,
V = Volume in ml of the final solution, and
M = Mass in g of the sample in the final solution.